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Transformation of Cl Bonding Structures on Si(100)-(2x1)

by

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Abstract

The digital electron stimulated desorption ion angular distribution (ESDIAD) method has been used to observe the existence and the irreversible thermal transformation between different bonding structures for Cl on the Si(100)-(2x1) surface. The ESDIAD pattern produced from dissociative chemisorption of Cl_2 at 120 K shows intense normal emission of Cl^+, plus lower intensity of Cl^+ emission focused along the [011] and [011] axes (the axes of Si_2 dimer orientations in two domains). Upon annealing ($T \leq 673$ K), the Cl^+-ESDIAD pattern is irreversibly transformed into a four-beam pattern with a two-fold azimuthal symmetry in the plane of each dimer axis, indicative of the formation of energetically stable Si-Cl bonds inclined 25°±4° from the surface normal.

Bonding configurations for atomic adsorbates on the Si(100) surface have received substantial attention, since the discovery of the 2x1 reconstruction on this surface.[1] Fundamental interest has been focused on (1) the bonding of atomic species with the highly directional surface dangling bonds,[2] and (2) surface restructuring induced by the adsorbate.[3] In chemisorption, the surface trades off energy lost by local bond formation with energy gained by elastic distortion of substrate atoms in search of the lowest free energy bonding configuration.

The bonding structure of chlorine on the Si(100) surface has been studied using various surface science techniques.[4-10] Conflicting results about this system exist in the literature. Based on the studies of photoemission spectroscopy and low-energy electron energy loss spectroscopy (LEELS),[4-6] it was suggested that two Cl atoms are bonded to the two silicon atoms comprising a symmetric Si_2 dimer with an off-normal Si-Cl bond. Near-edge X-ray absorption fine structure (NEXAFS) measurements were also interpreted to indicate that off-normal Si-Cl bonding occurs.[7] In these studies, the Si-Cl bond angle could not be determined. However, in contrast to earlier reports, a more recent NEXAFS study indicated that, following adsorption at 530 K, the Si-Cl bond is perpendicular to the (100) plane to within 10° (the experimental accuracy).[8,9] In conjunction with surface extended X-ray absorption fine structure (SEXAFS) measurements, Cl was postulated to chemisorb to a single Si atom in a buckled (asymmetric) Si_2 dimer with a normally-oriented Si-Cl bond.[9] This is inconsistent with the previously proposed off-normal Si-Cl bonding to a symmetric Si_2 dimer site.[4-7] Recent ESDIAD studies of Cl on Si(100) have proposed the thermal (300 K - 620 K) conversion of dichloride species to the normally-oriented monochloride species on asymmetric Si_2 dimer sites.[10]

In this work, we present an ESDIAD study of chlorine chemisorption on the Si(100) surface. ESDIAD images the surface-adsorbate bonding direction in surface species being dissociated by electron impact.[11] Evidence is presented here for the existence of two different bonding structures for Cl on Si(100) depending on the chemisorption conditions. The off-normal Si-Cl bonding geometry is identified as a lower-energy configuration for Cl chemisorbed on the Si(100) surface. This result sheds new light on the adsorbate bonding configuration on the Si(100) surface, and refines the NEXAFS measurement previously reported.[8,9]

Details of the ultrahigh vacuum (UHV) system (with a base pressure of $3x10^{-11}$ Torr) and the Si(100) crystal preparation have been described previously.[12] The UHV system is equipped with a CMA Auger electron spectrometer (AES), an argon ion sputtering gun, a digital LEED/ESDIAD apparatus, a shielded quadrupole mass spectrometer (QMS) for line-of-sight thermal desorption studies, and a second QMS with an attached electron gun for ion mass analysis in electron stimulated desorption (ESD). The Si(100) crystal (orientation accuracy= ±1°; 15x15x1.5 mm; p-type; B-doped; 10 Ω-cm) is resistively heated (120-1200 K) by a Honeywell programmable temperature controller used to drive a feedback circuit to control the heating power.[13] Dosing of Cl_2 was done by using a multicapillary-collimated doser in which the absolute flux of Cl_2 molecules onto the crystal was calibrated.[14] The identity of Cl^+ produced from ESD on Cl/Si(100) was determined by the auxillary QMS with the ionizer turned off. All ESDIAD measurements were made at 120 K and with an electron energy of 120 eV. For all the data presented here, a +10.0 V crystal bias potential was used to compress the ion trajectories. The digital ESDIAD data have been smoothed using a two-fold symmetrization procedure as described previously.[15] Small ion optical aberrations are removed by the azimuthal symmetrization method which assumes, based on the two-fold symmetry of the Si(100)-(2x1) surface, that Cl^+ ion intensities originating from chemisorbed Cl at a given polar angle and in azimuthal directions 180° apart should be equal.

Figure 1 shows the Cl^+-ESDIAD patterns before and after annealing a Cl-covered Si(100) surface. Figure 1(a) is obtained after dissociative chemisorption[16] of Cl_2 on the Si(100) surface at 120 K. The pattern is dominated by normal emission of Cl^+; there is also a lower intensity Cl^+ signal emitted off normal in the vertical planes parallel to the two dimer axes. In contrast, after chemisorption of Cl_2 on an Ar^+-roughened Si(100) surface there is no evident anisotropic distribution of intensity along the two dimer axes. Instead, a broad and isotropic normal Cl^+ beam characteristic of an average over a disordered surface was observed. This indicates that the pattern shown in Figure 1(a) is evidence for surface ordering on the Cl-covered Si(100) surface. Furthermore, the geometry of the pattern obtained on the ordered Si(100) surface is independent of Cl coverage, indicating that features of the ESDIAD pattern are not due to the preferential Cl adsorption onto surface defect sites. For all the Cl coverages, the two domain (2x1) LEED pattern is preserved upon chemisorption, showing that the Si(100)-(2x1) reconstruction is retained.[16]

An irreversible transformation of the Cl^+ ESDIAD pattern is observed upon annealing. The resulting Cl^+-ESDIAD patterns obtained at 120 K after annealing to 423 K and 673 K for 60 s are shown in Figure 1(b) and 1(c), respectively. The development of intensity in four Cl^+ beams oriented along the two dimer axes of the Si(100) surface, coupled with the decrease of the normal Cl^+ emission, can be seen as the temperature of the crystal is gradually increased. After annealing to 673 K, no prominent central beam remains. Further annealing to T > 673 K will cause a change in surface Cl coverage due to the etching of the Si surface via $SiCl_2$(g) desorption,[16,17] and thus, is beyond the scope of discussion in this report.

It is known that a Si(100) surface contains two types of neighboring terrace domains in which the orientations of the Si_2 dimer bonds are orthogonal to each other.[18] Thus, for a Si(100) surface containing these two domains, off-normal Si-Cl bonds lying in the vertical plane containing a dimer bond axis will produce a four-beam Cl^+ ESDIAD pattern. The four-beam pattern is a consequence of the superposition of a pair of two-fold azimuthal symmetric patterns originating from the two perpendicular domains. In addition, it can be seen from both the three dimensional ESDIAD plot (Figure 1, left) and the contour plot (Figure 1, right) that the Cl^+ emission intensity in the four beams are approximately the same. This indicates that the Si(100) crystal surface possesses nearly equal density of the two domains.[18]

A plot of Cl^+ intensity versus desorption angle is shown in Figure 2 (a cross-section view in a plane parallel to one of the dimer axes in Figure 1). Figure 2(a) shows a normal beam of Cl^+ with two shoulders symmetrically distributed with respect to the surface normal. Figures 2(b) and 2(c) show the attenuation of the relative intensity of the normal beam and the concomitant enhancement of the two off-normal Cl^+ beams as a consequence of heating to 423 K and 673 K, respectively. Analysis of the four-beam ESDIAD pattern as a function of crystal bias indicates that the peak maximum of Cl^+ emission has a polar angle of 28° ± 3° under a field-free condition.[19] Correction of this angle for image charge and reneutralization effects[20-22] leads to a Si-Cl bond angle inclined 25° ± 4° to the surface normal.[16] This estimated Si-Cl bond angle is in good agreement with general expectations based on the tetrahedral bonding structure of silicon. These results indicate that (1) the four-beam pattern is a result of single Cl atoms bonded to each dangling bond on the dimer, which is generally expected to be the lowest energy configuration between the adsorbate and the clean Si(100) surface,[2,3] and (2) the low-temperature structure produces a combination of both normal and inclined Cl^+ emission, and thus, represents a co-existence of two Cl bonding structures on Si(100).

A quantitative determination of the populations of the two Cl bonding structures can not be made due to the unknown ESD cross sections for the two structures, though a qualitative picture of the interconversion can be obtained from the beam peak intensity behavior. In Figure 3, the ratio of the peak height for the normal beam (H_N) to the peak height for the inclined beam (H_I) is plotted against the annealing temperature. Each data point is a result of annealing for 60 s of a Cl-covered surface at the indicated temperature. Longer annealing time does not significantly affect the ratio of H_I to H_N. Therefore, each data point shown in Figure 3 represents the essential completion of the structural conversion process at that certain temperature. The ratio of H_N/H_I decreases with increasing annealing temperature. The observed temperature dependence indicates that the structural transformation is a thermally activated process, readily observable at a temperature as low as ~ 273

K, but is not complete until ~ 600 K. This suggests that the structural transformation process initially has a low-energy barrier. However, the fact that the transformation occurs over a broad temperature range implies that a single kinetic process, involving a constant activation energy and preexponential factor, is not appropriate for describing the structural conversion process.

A possible mechanism for the structural transformation involves a change of adsorbate-substrate bonding coordination number, as schematically shown in Figure 4. The Cl atom is known to be able to form multiple-coordinated compounds in inorganic complexes.[23] Generally seen is the bridge-bonded, dicoordinated structure, e.g., as in Al_2Cl_6.[24] A bridge-bonded Cl on the Si(100)-(2x1) surface (Figure 4(a)) will require the simultaneous rupture of two Si-Cl bridge bonds during the ESD process to form Cl^+ which is predicted to escape in a normal direction from the surface.[25] A bridge-bonded Cl to two Si atoms has also been proposed for an adduct of a Cl atom on tetramesityldisilene in the gas phase.[26] Annealing of the Cl-covered Si(100) surface to higher temperatures causes the bridge-bonded Cl atom to move to the inclined dangling bond site, producing a more stable, monocoordinated Si-Cl bond (Figure 4(b)). As a consequence, the off-normal Si-Cl bonding configuration and two-fold azimuthal symmetry for each domain are observed. The observed structural transformation therefore indicates that the bridging Cl geometry is energetically less stable compared to the monocoordinated Si-Cl structure. Factors possibly influencing the energy are (1) the lack of direct interaction between the Cl atom and the surface dangling bond in the bridging geometry, and (2) relaxation of the strain on the Si-Si dimer bond in the inclined geometry (the monocoordinated structure).

Tilted Si-F bond have been observed on Si(100) by ESDIAD.[2a,2b] Theoretical studies of the chemisorption of fluorine on Si(100), using first principles electronic structure calculations, have suggested that both the bridge-bonded structure and the off-normal structure are stable, exhibiting different binding energies.[2(c)] The bond strengths of F on Si(100) for the dicoordinated, bridge-bonded structure (Si-F-Si) and the monocoordinated, inclined bonding structure (Si-F) are calculated to be 3.0 eV and 6.4 eV, respectively. However, it was also predicted that no barrier exists for the conversion of the bridging F atom to the monocoordinated F,[2(c)] which differs from the observed activated process associated with the structural transformation of Cl. The difference can be due to the greater diffuseness of the Cl 3p orbitals compared to the 2p orbitals of F. This leads to less strain in the bridge-bonded structure for Cl, because the equilibrium distance between Cl and the dimer bond will be larger than that of F. Using the semiempirical SLAB-MINDO method, both the bridge-bonded and monocoordinated structures have also been proposed upon dissociative chemisorption of Cl_2 and F_2 on

Si(100).[2(d)] However, in this case, the proposed bridge-bonded configuration involves the breaking of the Si-Si dimer bond, causing the formation of two Si-Cl bonds on the original dimer site. In addition, the predicted total energy for the bridge-bonded Cl is found to be 0.61 eV per surface atom lower in energy than the monocoordinated Cl. This is contradictory to our measurement which shows that the monocoordinated structure is certainly a lower energy configuration for Cl on the Si(100)-(2x1) surface.

In conclusion, evidence for the existence and the irreversible transformation of two bonding structures for Cl on the Si(100)-(2x1) surface is reported. This may explain conflicting results for the bonding structures of Cl on Si(100) previously presented in the literature.[4-10] Our measurements are the first to include Cl_2 adsorption on Si(100) below 300 K. They provide evidence for the co-existence of two bonding structures and an irreversible thermal conversion from one to the other. A normal ESDIAD beam of Cl^+, which is proposed to originate from a bridge-bonded Cl, is found at low temperatures originating from an intermediate binding state, while the four-beam pattern, derived from a Si-Cl bond inclined 25° ± 4° from the surface normal, represents a lower-energy configuration for the bonding of Cl on Si(100). The detailed kinetics for the transformation of local bonding structure are not understood at present; a single activated process with a constant barrier height is not appropriate to describe the conversion process.

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FIGURE CAPTIONS

FIGURE 1. ESDIAD patterns of Cl on Si(100) under the following conditions: (a) after dissociative chemisorption of Cl_2 at 120 K, with an exposure of nearly 1 ML (= 6.8×10^{14} Cl/cm^2), (b) and (c) subsequent annealing of the surface for 60 s to 423 K and 673 K, respectively. The figures on the left are perspective plots; they are generated by mapping the ion intensity into the z direction. The figures on the right are contour plots. Each contour line in each individual plot represents an increment of 1/6 of the plot's maximum. The width of the contours is ± 6 % of the median value stated for the contour.

FIGURE 2. Cl^+-ESD intensity versus polar desorption angle. The data are extracted from a cut along the [011] axis in Figure 1. The experimental conditions for each plot correspond to Figure 1. All the plots shown here have been normalized based on the maximum intensity being set equal.

FIGURE 3. The ratio of H_N to H_I versus the annealing temperature. H_N and H_I are defined in the text and Figure 2. The error bars indicated are obtained by comparing the data from two domains. The line joining data points is to aid the presentation.

FIGURE 4. Schematic pictures for the bonding configurations of Cl on Si(100): (a) the bridge-bonded structure, and (b) the inclined bonding structure.

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